

**(12) UK Patent Application (19) GB (11) 2 200 351 (13) A**  
(43) Application published 3 Aug 1988

(21) Application No 8702003

(22) Date of filing 29 Jan 1987

(71) Applicant

Leningradsky institut inzhenerov  
Zheleznodorozhnogo Transporta imeni Akademika  
V. N. Obratsova

(Incorporated in USSR)

Moskovsky prospekt 9, Leningrad,  
Union of Soviet Socialist Republics

(72) Inventors

Evgeny Georgievich Petrov  
Vitaly Sergeevich Dikarevsky  
Mikhail Spiridonovich Pavlov  
Nikolai Varfolomeevich Moskalov  
Ippolit Igorevich Stasjuk  
Sergel Mikhailovich Levitin

(51) INT CL<sup>a</sup>

C04B 35/18 C02F 1/28

(52) Domestic classification (Edition J):

C1J 10 14 25 4 9 X  
C1A 13 420 D31 N13 N4 VG1  
C1C 253 324 406 447 AH  
U1S 1356 1443 C1A C1C C1J

(56) Documents cited

None

(58) Field of search

C1J  
C1A  
C1C  
Selected US specifications from IPC sub-classes  
C02F C04B

(74) Agent and/or Address for Service

Mathisen Macara & Co  
The Coach House, 6/8 Swakeleys Road,  
Ickenham, Uxbridge, UB10 8BZ Middlesex

(54) **Process for producing filter material for water purification**

(57) A process for producing a filtering material for purification of naturally-occurring waters for the preparation of potable water comprises granulation of an aqueous suspension of "sapropel" in a fluidized bed, followed by calcination of the resulting granules at a temperature of 800-900°C inclusive.

"Sapropel" comprises slime-like deposits formed on the bottom of freshwater pools (lakes, ponds, swamps) from decayed vegetable and animal remnants, as well as from mineral admixes of a drift character. It is an aluminosilicate material containing a substantial proportion of organic matter (prior to treatment).

GB 2 200 351 A

PROCESS FOR PRODUCING FILTER MATERIAL  
FOR WATER PURIFICATION

The present invention relates to purification of naturally-occurring waters to obtain potable water and, more particularly, to processes for the production of a filtering material for purification of naturally-occurring waters.

Known in the art is a process for producing a filtering material for purification of naturally-occurring waters comprising granulation of a finely-divided naturally-occurring aluminosilicate raw material (natural clays) in a fluidized bed, followed by calcination of the resulting granules at a temperature of from 1,050 to 1,250°C (V.N.Martensen, R.I.Ayukaev, A.K.Strelkov, S.M.Shandalov, P.G.Bykova "Crushed Claydite-New Filtering Aid for Water-Purification Filters", Kuibyshev, 1976, pp.33-35).

The prior art process makes it possible to produce a filtering material which, however, exhibits insufficient adsorption properties. This is due to the fact that the surface of granules is fused to a great extent and has no open pores, it also features a low roughness, wherefore a specific surface area of such granules is small (about 1.0-1.2 m<sup>2</sup>/g).

Also known in the art is a process for producing a filtering material for purification of naturally-occurring waters comprising granulation of an aqueous suspension of a naturally-occurring aluminosilicate raw material (natural clays) in a fluidized bed with the addition of volcanic ashes or perlite, followed by calcination of the resulting granules at a temperature of from 1,000 to 1,250°C (S.P.Onatsky "Manufacture of Claydite", Izdatel'stvo Literaturny po Stroitel'stvu" (Construction Literature Publishing House),

Moscow, 1971, pp.122, 144).

The incorporation of volcanic ashes or perlite into the suspension slightly improves adsorption properties of the filtering material due to a certain increase of the specific surface area of the granules. However, this process results in the production of granules with a fused surface characterized by lack of open pores and by a low roughness, thus failing to ensure a substantial improvement of adsorption properties of the filtering material.

Furthermore, known in the art is still another process for producing a filtering material for purification of naturally-occurring waters comprising granulation, in a fluidized bed, of an aqueous suspension of a naturally-occurring aluminosilicate raw material (natural clays) with the addition of finely-divided magnetite in an amount of from 35 to 70% by mass of the clay, followed by calcination of the resulting granules at a temperature within the range of from 600 to 900°C (cf. USSR Inventor's Certificate No. 982723, Int.Cl.<sup>3</sup> B 01 D 23/10, published in the Official Bulletin "Discoveries, Inventions, Industrial Designs, Trademarks", No. 47, 23.12.82).

This process makes it possible to improve adsorption properties of the filtering material, since at the above-specified calcination temperature the granules get not fused, they have open pores and a higher specific surface area. However, an insufficient total volume of open substantially limits the possibility for increasing specific surface area and roughness of the granules. As a result, a filtering material with insufficient adsorption properties is obtained.

It is an object of the present invention to provide a process for producing a filtering material for purification of naturally-occurring waters which would make it possible to obtain a filtering material with a greater total volume of open pores and with a high specific surface area and, hence, with high adsorption properties.

This object is accomplished by a process for producing a filtering material for purification of naturally-occurring waters comprising granulation of an aqueous suspension of a naturally-occurring aluminosilicate material in a fluidized bed with a subsequent calcination of the resulting granules at a temperature of from 800 to 900°C, wherein, according to the present invention, as the naturally-occurring raw material sapropel is used.

The organic constituent incorporated in sapropel is burnt out upon calcination of sapropel which results in the formation of a porous structure of the granules with a high total volume of open pores. This, in turn, results in an increase in the specific surface area of the granules and in an improvement of absorption properties of the filtering material.

The filtering material produced by the process according to the present invention makes it possible to considerably improve effectiveness and quality of purification of naturally-occurring waters employed for the preparation of potable water.

As it has been already mentioned hereinbefore, the calcination of granules is carried out at a temperature within the range of from 800 to 900°C. It is inadvisable to conduct calcination of granules at a temperature of

less than 800°C, since at lower calcination temperatures the resulting granules have an insufficient mechanical strength. It is also inexpedient to carry out calcination of granules at a temperature above 900°C, since at higher  
5 temperatures of calcination the specific surface area of granules is reduced and a non-productive power consumption for calcination is increased.

In the process according to the present invention sapropel is used as the starting naturally-occurring aluminosili-  
10 cate raw material. Sapropel comprises slime-like deposits formed on the bottom of fresh-water pools (lakes, ponds, swamps) from decayed vegetable and animal remnants, as well as from mineral admixes of a drift character. The composition of sapropel is defined by conditions of its formation and  
15 can vary within a broad range.

For the production of a filtering material an aqueous suspension of sapropel is prepared at a mass ratio of the solid and liquid phases of from 1:1.5 to 1:2 respectively. The prepared suspension is granulated in an apparatus with  
20 a fluidized bed. The resulting granules are subjected to calcination at a temperature within the range of from 800 to 900°C and then cooled. The calcination of granules can be effected both in the same apparatus and outside it, for example in a calcination furnace.

25 As a result, a filtering material is obtained in the form of a mixture of granules with a diameter ranging from 0.5 to 2 mm.

The filtering material produced by the process according to the present invention can be effectively used in  
30 filtration facilities operating on the principle of both

ascending and descending filtration. It ensures a high quality of purification of naturally-occurring waters.

For a better understanding of the present invention, some specific examples illustrating its particular embodiments are given hereinbelow.

#### Example 1

For the production of a filtering material use is made of sapropel comprising slime-like deposits formed on the bottom of a fresh-water lake from decayed vegetable and animal remnants, as well as from mineral impurities of a drift character. Sapropel has the following chemical composition, % by mass:  $\text{SiO}_2$  - 34.1,  $\text{Al}_2\text{O}_3$  - 7.0,  $\text{Fe}_2\text{O}_3$  - 7.2,  $\text{CaO}$  - 3.5,  $\text{MgO}$  - 0.5, organic portion - 47.7.

The aqueous suspension of sapropel is prepared at the mass ratio of solid and liquid phases equal to 1:1.75 respectively. The resulting suspension is granulated in a fluidized-bed apparatus. The thus-obtained granules are discharged from the apparatus and supplied into a calcination furnace wherein they are subjected to calcination at the temperature of  $900^\circ\text{C}$ . Thereafter the granules are cooled.

As a result, a filtering material is obtained in the form of a mixture of granules with a diameter of from 0.5 to 2.0 mm.

#### Example 2

A filtering material is prepared in a manner similar to that described in Example 1 hereinabove. As the starting aluminosilicate raw material use is made of sapropel comprising slimy deposits formed on the bottom of a fresh-water lake from decayed vegetable and animal remnants, as well as from mineral impurities of a drift character. The che-

mical composition of sapropel is the following, per cent  
by mass:  $\text{SiO}_2$  - 39.4,  $\text{Al}_2\text{O}_3$  - 14.4,  $\text{Fe}_2\text{O}_3$  - 6.3,  $\text{CaO}$  - 7.6,  
MgO - 1.2,  $\text{K}_2\text{O}$  - 0.4, organic portion - 30.7. The mass  
ratio of sapropel to water in the suspension is equal to  
5 1:1.5 respectively. The temperature of calcination of gra-  
nules is  $800^\circ\text{C}$ .

#### Example 3

A filtering material is produced as described in Example  
1 hereinbefore. The mass ratio of sapropel to water in the  
10 suspension is equal to 1:1.75 respectively. The granules  
are subjected to calcination at the temperature of  $850^\circ\text{C}$ .

#### Example 4

A filtering material is produced in a manner similar to  
that described in Example 1. As the starting aluminosilicate  
15 raw material sapropel is used comprising slime-like deposits  
formed on the bottom of a fresh-water lake from decayed vege-  
table and animals remnants, as well as from mineral impurities  
of a drift character. The chemical composition of sapropel  
is the following, per cent by mass:  $\text{SiO}_2$  - 39.4,  $\text{Al}_2\text{O}_3$  - 14.4,  
20  $\text{Fe}_2\text{O}_3$  - 6.3,  $\text{CaO}$  - 7.6, MgO - 1.2,  $\text{K}_2\text{O}$  - 0.4, organic por-  
tion - 30.7. The mass ratio of sapropel to water in the sus-  
pension is equal to 1:2 respectively. The temperature of  
calcination of granules is  $900^\circ\text{C}$ .

Given hereinbelow in Table 1 are main characteristics  
25 of the filtering material produced in Examples 1 to 4 and,  
for the purpose of comparison, similar characteristics of  
the filtering material produced by the process of the USSR  
Inventor's Certificate No. 982723. Use is made of filtering  
materials with the fraction of granules of a diameter from  
30 1.0 to 1.2 mm. As the basic characteristics of the filtering

materials shown in the following Table are: total volume of open pores, specific surface area of the granules, adsorption capacity thereof. These characteristics are determined following generally accepted procedures described in the book by N.F. Ermolenko and M.D. Efros "Regulation of Porous Structure of Oxide Adsorbents and Catalysts", Minsk, 1971. The adsorption capacity is determined by adsorption of methylene blue dye-stuff.

Table 1

IO	Filtering material of	Total volume of open pores, cm <sup>3</sup> /g	Specific surface area of granules, m <sup>2</sup> /g	Adsorption capacity, mg.equiv/g
	Example 1	0.268	12.142	0.0321
I5	Example 2	0.214	11.085	0.0234
	Example 3	0.279	12.420	0.0332
	Example 4	0.221	11.335	0.0241
	USSR Inventor's Certificate			
20	No. 982723	0.097	3.063	0.0164

As it is seen from the foregoing Table 1, the filtering material produced by the process according to the present invention (Examples 1 through 4) is superior over the filtering material produced by the process according to the USSR Inventor's Certificate No. 982723 by 2.2-2.7 times in the total volume of open pores, by 3.6-4 times - in the specific surface area and by 1.5-2 times - in the adsorption capacity. Owing to the above-mentioned advantages, the filtering material produced by the process according to the



present invention can be successfully employed for an effective purification of naturally-occurring waters for the preparation of high-quality potable water.

The filtering material produced by the process according to the present invention and the filtering material produced according to the USSR Inventor's Certificate No. 982723 were tested in purification of river water having the following characteristics: turbidity - 7 mg/l, colour index - 41 degrees, pH = 7.1. The tests were carried out on an experimental stand consisting of two filtering columns of 500 mm diameter and 2,000 mm height. One of the columns is packed with the filtering material produced by the process according to the present invention, the other - with the filtering material produced according to the USSR Inventor's Certificate No. 982723. In doing so, the filtering materials were used in the fraction thereof having granules of a diameter 1.0-1.2 mm. The height of the bed of each filtering material in the column is 1,000 mm. The filtration of river water was effected in the descending direction. The filtration rate was kept constant and equal to 8 m/h. Directly prior to the supply of river water to the columns, a coagulation agent - aluminium sulphate was added to the water in the amount of 7 mg/l as calculated for  $Al_2O_3$ .

The criterion of efficiency of operation of the filtering materials was the amount of purified river water for one filtering cycle. The filtering cycle means a period of time after which a purified water goes out of a filtering columns; this water has to meet the requirements imposed on quality of potable water as to its turbidity and colour index, namely: turbidity - max. 1.5 mg/l, colour index - max. 20 degrees.

The results of process tests of the filtering materials produced by the process according to the present invention and by the process of the USSR Inventor's Certificate No. 982723 are shown in Table 2 hereinbelow.

5 It follows from Table 2 that the duration of one filtering cycle and, hence, quality of river water purified over one filtering cycle on a filtering column charged with the filtering material according to the present invention is superior by more than 2 times to the same characteristics obtained in a filtering column with the charge of the filtering material produced by the process according to the USSR Inventor's Certificate No. 982723.

Table 2

15	Process characteristics	Filtering material	
		Produced by the process of this invention	Produced according to USSR Inventor's Certificate No.982723
	Duration of one filtering cycle, hours	13	6
20	Quality of the filtrate during the filtering cycle: colour index, degrees	12-15	15-20
25	turbidity, mg/l	1-1.5	1-1.5
	Amount of river water purified during one filtering cycle, litres	203	94

30 Therefore, the process according to the present invention makes it possible to produce a filtering material with improved adsorption properties which, in turn, enables a substantial increase in the efficiency of operation of fil-

tering facilities for purification of naturally-occurring waters and provides a high-quality potable water.

CLAIMS

1. A process for producing a filtering material for purification of naturally-occurring waters comprising granulation of an aqueous suspension of sapropel in a fluidized bed, followed by calcination of the resulting granules at a temperature within the range of from 800 to 900°C.

2. A process for producing a filtering material for purification of naturally-occurring waters according to the foregoing Claim 1, substantially as described in the Specification and Examples 1-4 given hereinbefore.

3. A filtering material for purification of naturally-occurring waters, whenever produced by the process according to the foregoing Claims 1 and 2.